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RAMAN SPECTRA OF HIGHLY CONDUCTING POLY-P-PHENYLENE COMPLEXES. (U)

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by

Costas H. Tzinis, Ray H. Baughman, and William M. Risen, Jr.

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Raman Spectra of Highly Conducting Poly-p-phenylene Complexes

by

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Abstract

The resonance Raman spectra of poly(p-phenylene) (PPP) and the highly conducting materials formed by doping it with the electron-donor Na (Na-PPP) or the electron acceptor AsF_5 (AsF_5 -PPP) have been obtained. Doping with either type of dopant causes the C-C bond joining the phenyl-rings to become stronger and at least one of the in-ring bonds to become weaker, so all C-C bonds along the backbone of the π -electron system become more alike than they are in the undoped PPP. The resonance enhancements show the relationship between changes in molecular configuration caused by optical transitions in the donor- or acceptor-doped PPP materials and the vibrational modes of the polymer.

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Introduction

The discovery of several highly conducting polymer systems, including charge-transfer complexes of poly(p-phenylene) (PPP) (1,2) and of polyacetylene (PA) (3,4), has attracted a great deal of attention. Through chemical doping with electron-acceptors, such as AsF_5 , or donors, such as Na, the electrical conductivity of these conjugated π -electron systems can be increased from their intrinsic insulating values of less than $1 \times 10^{-10} \text{ S m}^{-1}$ to metallic values of greater than $1 \times 10^3 \text{ S m}^{-1}$. Despite numerous studies of the properties of these materials, relatively little is known about the nature of the polymer chain after doping (5).

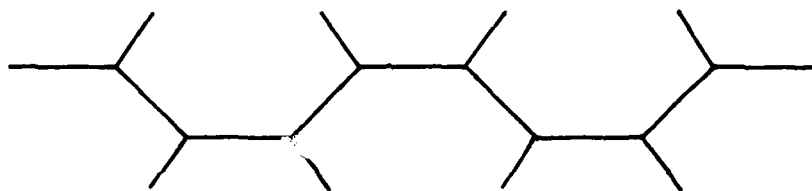
We report that the resonance Raman spectra of poly(p-phenylene) and its highly conductive AsF_5 - and Na-doped charge transfer complexes reveal the effects on the polymer caused by doping.

The structure of PPP is shown in Fig 1 (1). We assume, as in studies of PA- and graphite-complexes, that the basic PPP structure remains in the doped PPP. This result has been confirmed by investigations on AsF_5 - and Na-doped PPP (1,2). However, since the dopants transfer electrons onto the polymer chain (electron donor complexes, electron carriers) or away from the chain (electron acceptor complexes, hole carriers), it is expected that the strengths and lengths of certain of the in-chain C-C bonds are changed by doping. Such changes in carbon-carbon bond strength should cause changes in the Raman-observable bond-stretching frequencies. And, of course, the directions and magnitudes of the frequency shifts should reflect the changes in electronic structure of the chains.

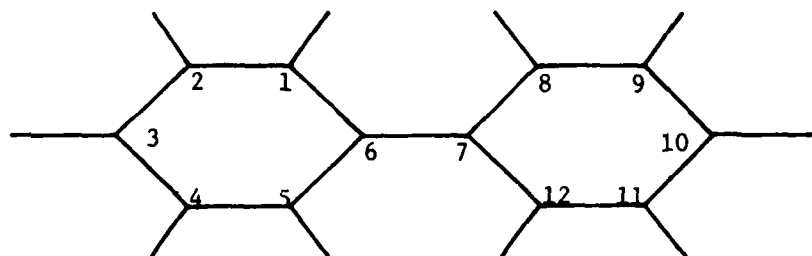
Experimental

The poly(p-phenylene) used in the present study was synthesized using

COMPARISON OF PA AND PPP LOCAL STRUCTURES



CIS-POLYACETYLENE (CIS-PA)



POLY-P-PHENYLENE (PPP)

Figure 1

the Kovacic synthetic method, as previously reported (1), and consists primarily of uncrosslinked para-linkages. It was prepared into cylindrical pellets. Elemental analyses of the samples showed their C to H atomic ratios to be 1.48-1.50, which are in good agreement with the value of 1.5 expected for $(C_6H_4)_x$. Acceptor doping was accomplished by exposing the PPP pellets to 450 Torr AsF_5 , under reaction conditions described previously, which resulted in green-black materials with 0.32 ± 0.08 moles AsF_5 per mole of monomer units $[C_6H_4(AsF_5)_{0.32}]_x$ and F to As atomic ratios of $4.75-5.10 \pm 0.30$ and C to H ratios of 1.5. Neutron activation analysis of similar pellets indicated a dopant concentration near the surface of $[C_6H_4(AsF_5)_{0.42}]_x$, and that in the interior of $[C_6H_4(AsF_5)_{0.24}]_x$. Electron donor doping was accomplished by treating PPP with sodium naphthalide in tetrahydrofuran solution, resulting in gold-appearing materials. The doped materials were handled in vacuo or in an argon-filled glove box and were sealed in a glass vial for spectroscopic study. The conductivity of identically AsF_5 -treated PPP was $\sigma \approx 1.7 \times 10^4 \text{ S m}^{-1}$; those of Na-PPP approach $1 \times 10^3 \text{ S m}^{-1}$.

The Raman spectra of PPP (spinning pellet) and doped PPP (Na-PPP, and AsF_5 -PPP) (samples sealed in glass tubes) were investigated using 632.8 nm (Spectrophysics 125) and 488.0 nm (Spectrophysics 165) laser radiation and a Jarrell-Ash 25-300 Raman spectrometer operating at 2 cm^{-1} resolution.

Results

The Raman spectra of PPP, Na-PPP, and AsF_5 -PPP obtained with the 632.8- and 488.0-nm laser sources are shown in Figs 2 and 3. The positions of three principal bands are tabulated in Table 1, and the forms of the vibrations to which they are assigned are illustrated by analogy to those of biphenyl (6) in Fig 4. Note that the arrows give direction and relative amplitude information only.

RAMAN SPECTRA OF PPP AND ITS DERIVATIVES (LASER WAVELENGTH: 6328 Å)

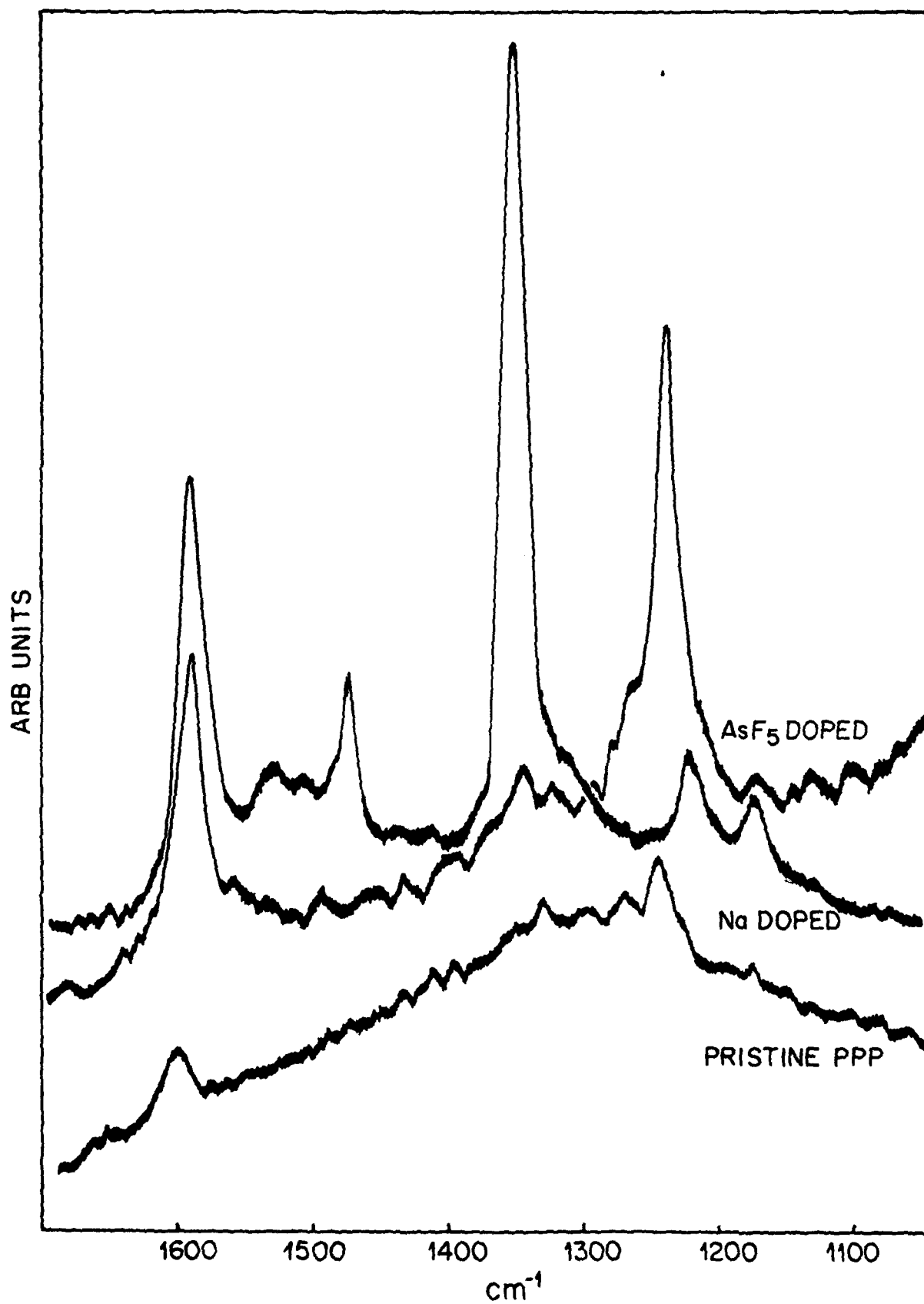


Figure 2

RAMAN SPECTRA OF PPP AND ITS DERIVATIVES (LASER WAVELENGTH: 4880 Å)

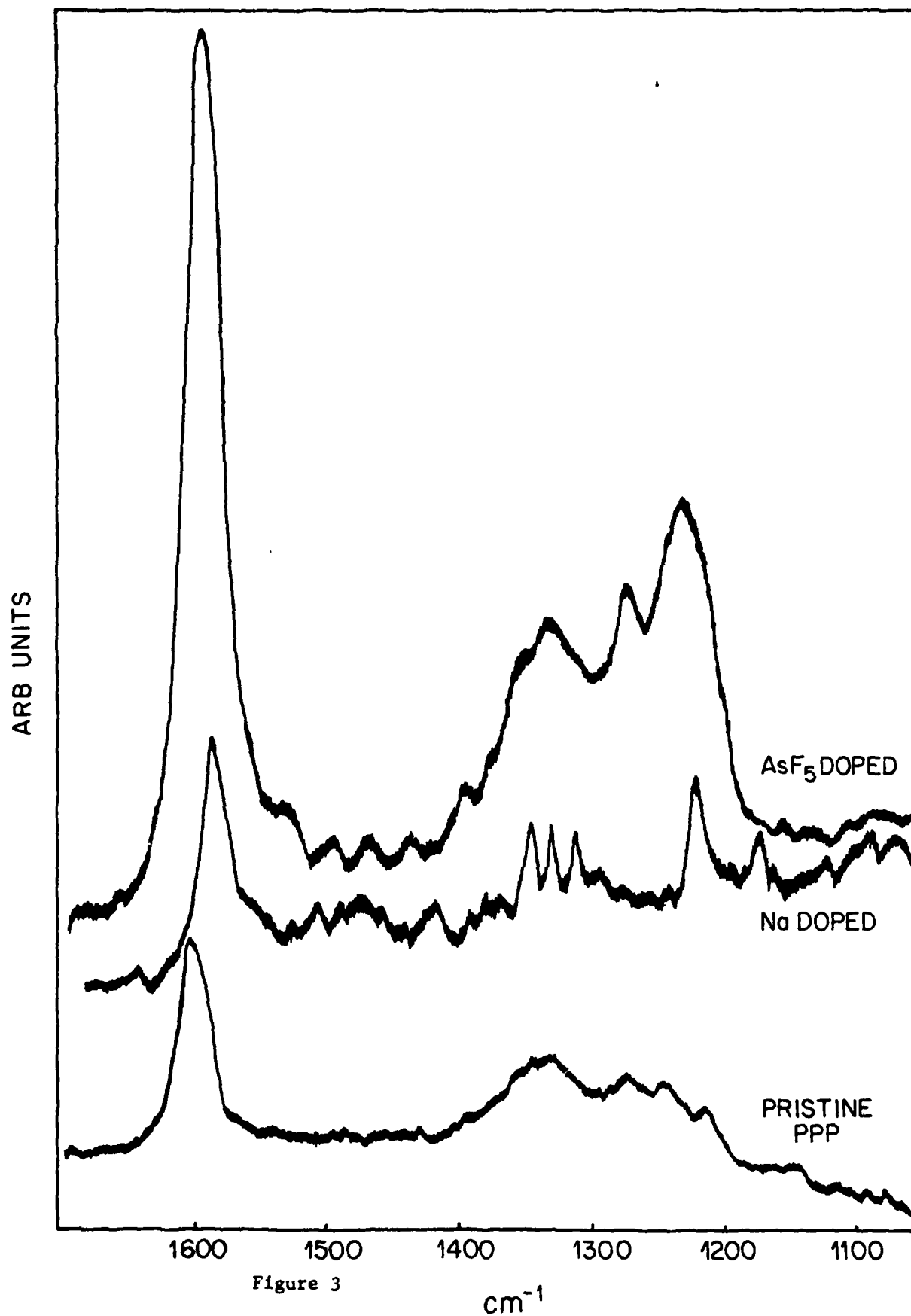


Table 1

Observed Raman bands assigned to ν_1 , ν_2 , and ν_3 of PPP and doped-PPP

Raman source	Material	$\nu_1 (\text{cm}^{-1})$	$\nu_2 (\text{cm}^{-1})$	$\nu_3 (\text{cm}^{-1})$
488.0 (nm)	PPP	1605	1337	1247
488.0	Na-PPP	1591	1350	1232
488.0	AsF ₅ -PPP	1588	1352	1223
632.8	PPP	1601	1330	1245
632.8	Na-PPP	1590	1342	1239
632.8	AsF ₅ -PPP	1588	1346	1222

THREE TOTALLY SYMMETRIC (A_g) VIBRATIONS
OF THE $-C_{12}H_8-$ REPEAT UNIT IN PPP

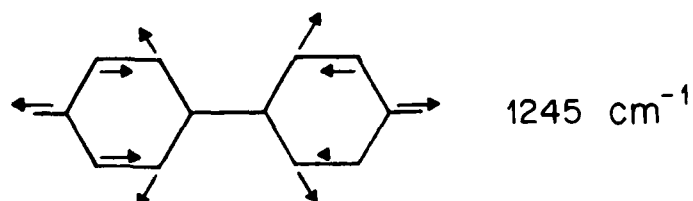
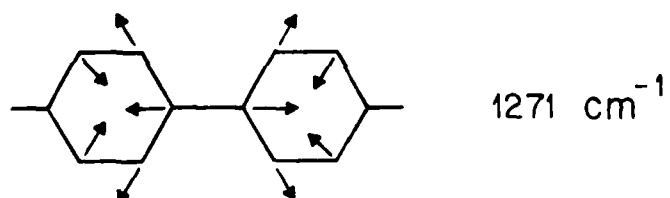
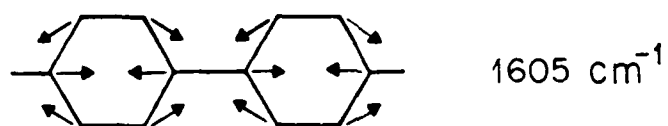


Figure 4

Interpretation of the resonance Raman observations requires knowledge of the optical absorption spectra of PPP and doped PPP in the region of the Raman source frequencies. The most useful available information of this type (2) is given in Fig 5, which shows the spectrum of doped polymerized *p*-terphenyl, which is an acceptor-doped highly conducting poly(*p*-phenyl) polymer, and of *p*-terphenyl itself. In reporting these spectra, Shacklette *et al.* noted that the high frequency absorption (near 3.6 eV) is that for a $\pi\text{-}\pi^*$ transition of the chain, and is essentially the same for the AsF_5 -doped or $(\text{CH}_3)_2\text{NH}$ -compensated material, while the lower frequency band, peaking below 1 eV (specifically 3600 cm^{-1}), vanishes when the AsF_5 -doped material is compensated. The two vertical arrows mark the frequencies of the Raman sources.

It is also useful in interpreting the Raman spectra to note the results of molecular orbital (Hückel SCF-MO) calculations on biphenyl and related poly(*p*-phenyls) (7). Much of the information required is contained in the symmetry properties of the orbitals and for the undoped PPP such properties are represented well (and simply) by the orbitals on biphenyl. Of the twelve π -electron SCF-MO's computed using the C 2p atomic orbitals as the basis set, the key ones for the present purpose are ψ_3 and ψ_{10} , whose energy levels are represented in Fig 6, and, particularly, ψ_3 , ψ_6 , ψ_7 , and ψ_{10} , whose symmetry properties are represented by the Hückel coefficients in Table 2. In Table 2 the coefficients C_{ij} of the 2p orbitals, ψ_j , normal to the plane on each of the twelve carbons as numbered on Fig 1, in the molecular orbitals, ψ_i are listed. Thus, C_{ij} for $\psi_i = \sum_j C_{ij} \phi_j$, are listed. Coefficients of the same sign on adjacent atoms designate a net bonding effect, and when they are of opposite sign a node appears between the atoms and the net effect of occupancy of the orbital in an antibonding contribution to the interaction of these two atoms. Identical sets of absolute values of coefficients

Table 2

Hückel coefficients, C_{ij} , for biphenyl

$j \backslash i^*$	3	6	7	10
1	.06	.30	.30	.06
2	-.29	-.14	.14	.29
3	-.44	-.40	-.40	-.44
4	-.29	-.14	.14	.29
5	.06	.30	.30	.06
6	.37	.37	-.35	-.37
7	.37	-.35	-.35	.37
8	.06	-.30	.30	-.06
9	-.29	.14	.14	-.29
10	-.44	.40	-.40	.44
11	-.29	.14	.14	-.29
12	.06	-.30	.30	-.06

*The coefficients C_{ij} in the equation, $\psi_i = \sum_j C_{ij} \phi_j$, are listed. Here the ψ_j are the C 2p orbitals normal to the biphenyl plane, numbered as shown in Figure 1, and the ϕ_i are numbered as shown in Figure 6 and in reference (7), from which the results are abstracted.

on certain sets of orbitals, e.g. those of ψ_6 and ψ_7 and those of ψ_3 and ψ_{10} , are symmetry required.

While it is not strictly valid to assume that addition or subtraction of an electron from the π -electron system of either PPP or biphenyl leaves its one-electron eigenstates unchanged, both spectral (8,9) and theoretical (10,11) studies of the biphenyl radical anion indicate that the effects on vibrational and electronic transitions are as would be expected if an electron were added to ψ_7 of the parent biphenyl. This result clearly is only an approximation, but using it in the present case of doped PPP provides a useful framework in which to describe an interpretation of the observed Raman spectra.

Discussion

The Raman spectra yield two main types of information. The first is the effect of doping on the vibrational frequencies of the polymer. As shown in Table 1, ν_1 and ν_3 decrease and ν_2 increases on doping. As illustrated by the forms of the vibrations in biphenyl, this means that doping causes the bridge C-C bond (C_6-C_7 in biphenyl) to become stronger and the in-ring C-C bonds (e.g. C_2-C_1 and C_1-C_6 in biphenyl) to become weaker. Thus, doping with an electron-donor or with an electron-acceptor causes essentially the same effect. While the magnitudes of the shifts differ slightly (note that the dopant levels are not identical), the directions are the same for both types of dopants.

These results are qualitatively consistent with the conclusions that would be drawn from the assumption that the one-electron eigenstates of the π -system with an electron added or subtracted are quite similar to those of the undoped material. That is, that the effect on bond strengths of adding an electron to the π -system (in a ψ_7 -like orbital) would be substantially the same as removing one (from a ψ_6 -like orbital). For example, as can be seen from the coefficients in Table 2, The C_6-C_7 interaction in ψ_6 is antibonding, so removal of an electron should make it stronger, while the C_6-C_7 interaction in ψ_7 is bonding so addition of

an electron to it should also make the C_6-C_7 bond stronger. Similarly, both types of in-ring C-C bonds should become weaker as they do.

The resonance Raman effects in the spectra also aid in understanding the bonding changes in the π -electron system that accompany doping. As shown in Fig 2, the ν_1 band (ca 1600 cm^{-1}) is of appreciable intensity in all three materials when the Raman source wavelength is 488.0 nm. This Ag mode is expected to be strong. Since it is relatively stronger in the 488.0 nm spectrum than in the 632.8 nm spectrum, it is assigned as being preresonantly enhanced through coupling with the optical transition at ca 3.6 eV.

The striking intensity enhancements are of ν_2 in Na-PPP and ν_3 in AsF_5 -PPP with 632.8 nm excitation. These are resonantly enhanced by their coupling with electronic transitions giving rise to a lower frequency optical absorption in either of the doped materials. In the case of AsF_5 -PPP this transition leads to the strong 0.6-2.0 eV band in Fig 5. In the Na-PPP case a similar low frequency band will be due to similar transitions, but in the π^* band.

The electronic transition of Na-PPP excited at 632.8 nm gives rise to electron density changes and a consequent molecular distortion that causes the ν_2 vibration to be resonantly enhanced. A similar observation in the biphenyl and biphenyl-anion (BP^-) system was shown by Yamaguchi, et al.(10) to be due to the fact that the changes in the equilibrium bond lengths of BP^- associated with the ψ_7 to ψ_{10} transition is of the same form as ν_2 . Assuming the analogy between BP and PPP and between BP^- and Na-PPP to hold at least insofar as their symmetry properties are concerned, the resonance enhancement of ν_2 in Na-PPP can be understood. Using it, the 632.8 nm Raman source would excite a transition from ψ_7 -like to a ψ_{10} -like orbital. The electron density changes accompanying that transition affects most the C-C bond that joins the rings, affects C_1-C_6 -like bonds significantly and C_2-C_1 bonds very little.

ELECTRONIC SPECTRA BEFORE AND AFTER DOPING
OF PA AND PPP

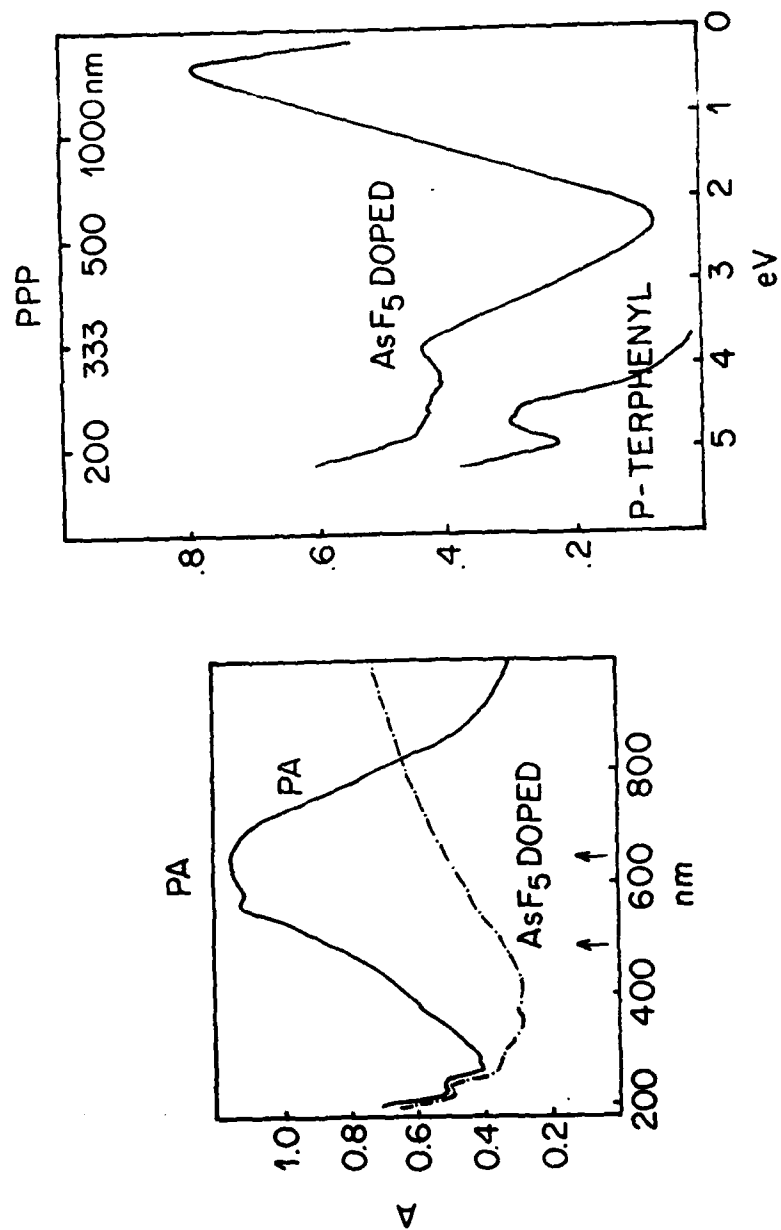


Figure 5

ALLOWED ELECTRONIC TRANSITIONS,
IN THE VISIBLE, OF A SIMPLE TWO
RING SYSTEM (BIPHENYL)

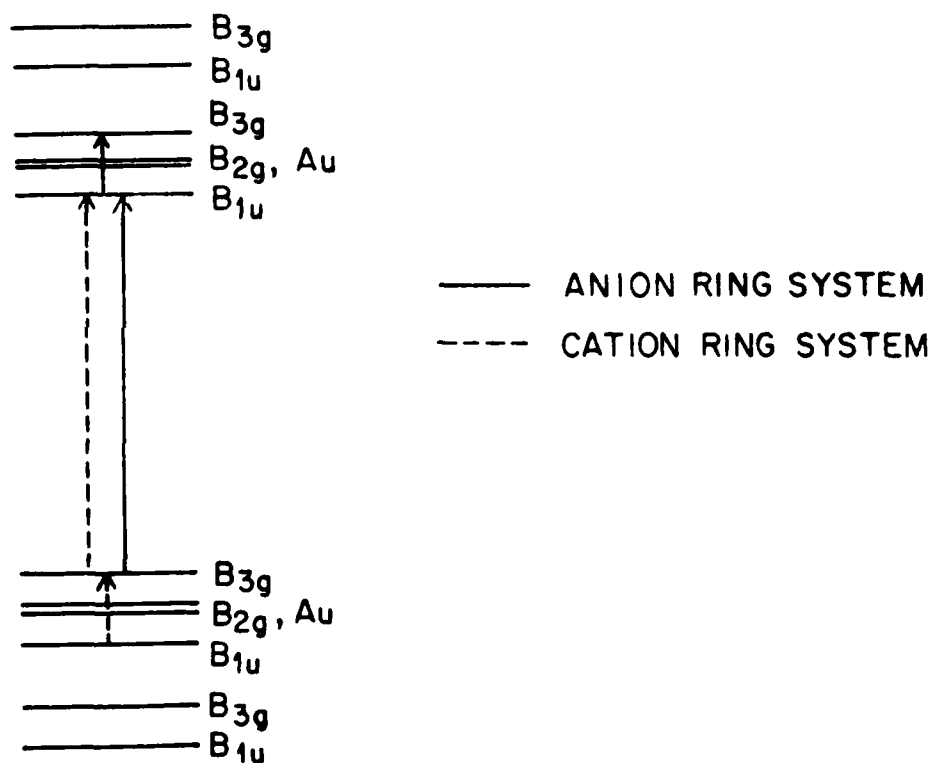


Figure 6

Similar arguments lead to describing the low frequency transition in $\text{AsF}_5\text{-PPP}$ as being that between ψ_3 -like and ψ_6 -like orbitals, which is possible because AsF_5 has removed an electron from the ψ_6 -like orbital. The electron-density changes accompanying this transition primarily affect the in-ring C-C bonds, do not affect the C-C bonds that connect the rings, and enhance ν_3 predominantly.

While it is helpful in interpreting and discussing the Raman spectra to use the terms provided by the Hückel calculations, it is important to note the conclusions that can be drawn on the basis of the Raman results alone. First, the bridge carbon-carbon bond (analogous to $\text{C}_6\text{-C}_7$) becomes stronger (and presumably shorter) while at least one of the in-ring carbon-carbon bonds become weaker (and longer) when PPP is doped with either an electron donor or acceptor. Second, the electron density changes that result from the optical transitions excited at 632.8 nm causes distortions similar in form to ν_2 in the Na-PPP case and to that of ν_3 in $\text{AsF}_5\text{-PPP}$. These conclusions neither depend on nor demonstrate the validity of the Hückel description, but they are qualitatively consistent with it.

Overall, both the frequency shifts and the resonance enhancement behavior of the Raman bands for both acceptor- and donor-doped poly(p-phenylene) are in accord with the assumption that an electron is added to or taken from the π -system of the parent polymer. They are qualitatively in accord with the predictions that can be made by assuming molecular orbitals of the extended Hückel SCF-MO type and assuming that the one-electron eigenstates give a reasonable approximation to the states that obtain upon doping. More detailed analyses of the nature of the materials after doping must include the rearrangements affected by changes in electron population and bond length changes, of course, and will be reported later.

Acknowledgments

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11. See ref (5), in which the $\pi \rightarrow \pi$ and $\pi^* \rightarrow \pi^*$ transitions are calculated.

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